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IS 8397 (1976): 2-nitrotoluene [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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Indian Standard
SPECIFICATION FOR 2-NITROTOLUENE

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SPECIFICATION FOR 2-NITROTOLUENE

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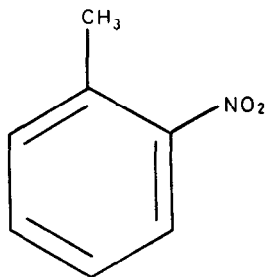
Indian Standard

SPECIFICATION FOR 2-NITROTOLUENE

0. F O R E W O R D

0.1 This Indian Standard was adopted by the Indian Standards Institution on 6 August 1976, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 2-Nitrotoluene ($C_7H_7O_2N$) is an important intermediate used in the manufacture of dyes and explosives. It is also known as 2-nitro-1-methyl benzene. It is obtained by nitration of toluene. It is represented by the following structural formula:



2-NITROTOLUENE
(MOLECULAR MASS 137)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 2-nitrotoluene.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of yellowish oil which on storage turns brownish.

*Rules for rounding off numerical values (revised).

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 2-NITROTOLUENE

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL NO. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Crystallizing point, °C, <i>Min</i>	-9.5	A-1
ii)	Assay, percent by mass, <i>Min</i>	99.0	A-2
iii)	3-Nitrotoluene, percent by mass, <i>Max</i>	0.2	A-2
iv)	4-Nitrotoluene, percent by mass, <i>Max</i>	0.2	A-2
v)	Dinitrotoluene, percent by mass, <i>Max</i>	0.1	A-3
vi)	pH value of aqueous extract	6.5 to 7.5	A-4

NOTE — 2-Nitrotoluene exists in two polymorphic forms, namely, alpha type crystals having crystallizing point of -9.5°C and beta type crystals having crystallizing point of -3.5°C. Generally the crystallizing point recorded is that of alpha form only. However, there are instances when there is another crystallizing point, namely, that of beta form. In such cases, the sample should be heated to its boiling point, cooled and then the crystallizing point redetermined. If the beta form persists, the crystallizing point determination should be further repeated by using seed crystals of some other sample that exists in alpha form.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (see IS:2552-1970*) or as agreed to between the purchaser and the supplier. The containers shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

- Name of the material;
- Name of the manufacturer and his recognized trade-mark, if any;
- Batch number;
- Tare, net mass and gross mass; and
- The minimum cautionary notice worded as under:

‘TOXIC. AVOID INHALATION OF FUMES AND ANY CONTACT WITH SKIN. DANGER OF CYANOSIS.’

*Specification for steel drums (galvanized and ungalvanized) (first revision).

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS:5299-1969*.

4.2 Number of Tests

4.2.1 Tests for crystallizing point and assay shall be conducted on each of the individual samples.

4.2.2 Tests for the remaining characteristics, namely, 3-nitrotoluene, 4-nitrotoluene, dinitrotoluene and pH value of aqueous extract shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirements of crystallizing point and assay if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample (*see 4.2.2*), the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the method prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (*see IS:1070-1977†*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

(Table 1, and Clause 5.1)

METHODS OF TEST FOR 2-NITROTOLUENE

A-1. CRYSTALLIZING POINT

A-1.1 Determine the crystallizing point of the material as prescribed in 7 of IS: 5299-1969*.

A-2. ASSAY, 3-NITROTOLUENE AND 4-NITROTOLUENE

A-2.0 Outline of the Method — The contents of 2-, 3-, and 4-nitrotoluenes are determined by gas chromatographic analysis.

A-2.1 Apparatus

A-2.1.1 Gas Chromatograph — with flame-ionization detector.

A-2.1.2 Potentiometric Strip-Chart Recorder — full scale deflection 1 second.

A-2.1.3 Temperature Programmer

A-2.1.4 Column — made of stainless steel, 2 m long, 6 mm in diameter, and packed with 20 percent XE-60 on Chromosorb W (AW — HMDS)† or an equivalent material.

A-2.2 Reagents

A-2.2.1 2-Nitrotoluene

A-2.2.2 3-Nitrotoluene

A-2.2.3 4-Nitrotoluene

A-2.2.4 Monochlorobenzene — internal standard.

A-2.2.5 Toluene

A-2.2.6 Acetone — solvent.

A-2.3 Procedure

A-2.3.1 Operating Parameters of Gas Chromatograph

A-2.3.1.1 Injection port temperature — 205°C.

*Methods of sampling and tests for dye intermediates.

†These are imported chemicals and are generally available with the fabricators of gas chromatographs and also with the stockists of chemicals.

A-2.3.1.2 Column temperature — 135°C.

A-2.3.1.3 Carrier gas — nitrogen, 30 ml/min.

A-2.3.1.4 Chart speed — 5 mm/min.

A-2.3.2 Sample — Dissolve 1 g of the sample in acetone. Add a known mass of the internal standard and make up the volume to 10 ml with acetone. Inject 2 microlitres.

A-2.3.3 Calibration Factor — Internal standardization is the method used for calibration of the results of analysis. Make up and chromatograph known mixtures of component 'n' for which the calibration factor is to be determined together with the internal standard plus other components of the sample. Calculate the calibration factor as follows:

$$K_n = \frac{X_n \times A_s}{X_s \times A_n}$$

where

K_n = calibration factor for component 'n' against internal standard 's',

X_n = mass percent of component 'n' in the sample,

A_s = peak area for internal standard 's',

X_s = mass percent of internal standard 's' in the sample, and

A_n = peak area for component 'n'.

A-2.3.4 Interpretation of Chromatogram — Elution order of components is as follows:

- a) Acetone (solvent used),
- b) Toluene,
- c) Monochlorobenzene,
- d) 2-Nitrotoluene,
- e) 3-Nitrotoluene, and
- f) 4-Nitrotoluene.

Identification is done with the aid of test mixtures.

A-2.4 Calculation

$$\text{Component 'n' in the sample, } \frac{\text{percent by mass}}{\text{percent by mass}} = \frac{K_n \times A_n \times X_s}{A_s}$$

where

K_n = calibration factor for component 'n' (see A-2.3.3),

A_n = peak area for component 'n',

X_s = mass percent of internal standard added to the sample,
and

A_s = peak area for internal standard 's'.

A-3. DETERMINATION OF DINITROTOLUENE CONTENT

A-3.1 Apparatus

A-3.1.1 Photo-Colorimeter

A-3.2 Reagents

A-3.2.1 Acetone

A-3.2.2 Sodium Hydroxide Solution — 0.1 percent (m/v).

A-3.2.3 Dinitrotoluene — pure (reference sample).

A-3.3 Procedure — Take 1 g of the sample, dissolve it in acetone and make up the volume to 10 ml with acetone. Take 1 ml of this solution in a 25-ml standard flask, add 1 ml of sodium hydroxide solution and make up the volume with acetone to 25 ml. Leave it for 10 minutes at 25°C to develop maximum colour and measure the absorption of colour developed using yellow filter and blank solution as control. Obtain the dinitrotoluene content from the calibration curve (see A-3.4).

NOTE — Yellow filter corresponds to 590 nm.

A-3.4 Calibration Curve — Weigh exactly 1 g of pure dinitrotoluene and dissolve it in acetone. Make up the volume exactly to 250 ml in a volumetric flask. Take 1 ml therefrom and make it up exactly to 100 ml with acetone. Take 1 ml, 2 ml, 3 ml and 4 ml from this solution in separate 25-ml standard flasks, add 1 ml of sodium hydroxide solution to each and make up the volume with acetone to 25 ml in each flask. Allow the solution to develop maximum colour (about 10 minutes) and measure the absorption using yellow filter. Plot a graph showing concentration in mg versus absorbance.

A-4. DETERMINATION OF pH VALUE OF AQUEOUS EXTRACT

A-4.1 Procedure — Take 100 ml of the sample in a separating funnel, mix it with 100 ml of hot water previously heated to boiling and shake the mixture well. Rapidly cool the separating funnel to room temperature. Allow the layers to separate. Separate the aqueous layer and measure its pH by pH paper or pH meter.